LIST OF U.S. CUSTOMS LABORATORY METHODS

USCL NUMBER	METHOD	TITLE
11-01	AOAC 965.22	**Sorting Corn Grits** **Sieving Method - Modified**
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U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 11-01 INDEX

AOAC 965.22
Sorting Corn Grits
Sieving Method - Modified

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

Certain products of Chapter 11 of the Harmonized Tariff of the United States (HTSUS) must pass certain sieving requirements, Chapter Note 2(B). This method is provided to give general guidance in sieving, using the aperture sizes noted in Chapter Note 2(B).

2 REFERENCES

AOAC 965.22
Sorting Corn Grits
Sieving Method

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 11-02 INDEX

Starch Determination in Cereal Products Using the Modified Ewers' Polarimetric Method

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

In Note 2A(a), Chapter 11, HTSUS, it states that starch content of products from the milling of the cereals listed in Note 2, is determined by the modified Ewers' polarimetric method. The purpose of the method is to quantitatively determine the starch content of cereal products.

2 REFERENCES

Examination and Analysis of Starch and Starch Products
Radley, J.A.
Applied Science Publishers Ltd., London
1976, p. 171-175

Analytical Method of Starch
Central Customs Laboratory Method No. 6 (tentative) No. 15

A Critical Assessment of the Parameters Affecting the Official EC "Ewers" Method for the Determination of Starch
J.F. Kennedy, D.L. Stevenson, and C.A. White
Birmingham, England (United Kingdom)

An Objective Illustration of the Inadequacy of the Official EC "Ewers" Method for the Determination of Starch
J.F. Kennedy, D. L. Stevenson, and K. Junel
Birmingham, England (United Kingdom)

3 EQUIPMENT AND REAGENTS

3.1 Analytical balance capable of determining the mass of the specimen to an accuracy of ±0.0001 gram

3.2 125 mL Erlenmeyer flasks

3.3 100 mL volumetric flasks

3.4 Starch standards of wheat, maize (corn), oat, and rice

3.5 Hydrochloric acid (HCl), reagent grade 0.31 M (26 mL concentrated HCl in 1 L of distilled water)

3.6 Trifluoroacetic acid (TFA), reagent grade 0.30 M (23 mL in 1 L of distilled water)

3.7 Stirrer/hot plate

3.8 Sodium phosphotungstate solution, 4% (10 mL required per sample)

3.9 Distilled water

3.10 Filter paper #5

3.11 Saccharimeter

4 PROCEDURE

4.1 Preparation of standards

4.1.1 Weigh 1, 2, 3, 4, and 5 grams, to the nearest 0.001 gram of starch standard of the cereal to be tested into the 125 mL flasks.

4.1.2 Add 25 mL of 0.31 M HCl to the flask and shake well.

4.1.3 Slowly wash the neck of the flask with an additional 25 mL of 0.31 M HCl, add a stirring bar and stir for 2 minutes.

4.1.4 Place the flask in a beaker of boiling water on a stirrer/hot plate and stir for precisely 15 minutes ensuring that the water continues to boil for the entire 15 minutes.

4.1.5 Immediately cool to room temperature (20EC), either shaking in a water bath or in a sink of water constantly adjusted to 20EC.

4.1.6 Transfer the contents of the flask to a 100 mL volumetric flask, wash the flask with three 10 mL volumes of distilled water and add to the volumetric flask.

4.1.7 Add 10 mL of 4% sodium phosphotungstate at 20EC to the contents of the volumetric flask and swirl to mix.

4.1.8 Make up to 100 mL with distill water, cap and shake.

4.1.9 Filter through a #5 filter paper, discarding the first 15 mL of filtrate.

4.2 Preparation of samples

4.2.1 Weigh 5 grams, to the nearest 0.001 gram, of finely powder[1] sample into a 125 mL graduated flask.

4.2.2 Add 25 mL of 0.31 M HCl to the flask, cap and shake well.

4.2.3 Wash the neck of the flask with additional 0.31 M HCl and continue shaking for an additional 2 minutes, remove the cap.

4.2.4 Place the flask in a beaker of boiling water on a stirrer/hot plate and stir for precisely 15 minutes ensuring that the water continues to boil for the entire 15 minutes.

4.2.5 Immediately cool to room temperature (20°C), either shaking in a water bath or in a sink of water constantly adjusted to 20°C.

4.2.6 Transfer the contents of the flask to a 100 mL volumetric flask and wash the flask three 10 mL volumes of distilled water and add to the volumetric flask.

4.2.7 Add 10 mL of 4% sodium phosphotungstate at 20°C to the contents of the volumetric flask and swirl to mix.

4.2.8 Make up to 100 mL with distilled water, cap and shake.

4.2.9 Filter through a #5 filter paper, discarding the first 15 mL of filtrate.

4.3 Analysis of the standard and sample solutions

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[1] If the sample is not in powder form, grind in a Wiley Mill.

4.3.1 Immediately wash the flow through cell on the saccharimeter with 30 mL of the filtrate of each standard and sample prior to filling the cell for a reading. (the flow through cell requires approximately 50 mL of solution.)

4.3.2 Read each value off the saccharimeter.

4.4 Moisture Determination

4.4.1 Conduct Thermal Gravimetric Analysis (TGA) on each standard starch to determine the temperature at which the sample loses moisture.

4.4.2 Dry, to the nearest 0.001 grams, 2 grams of each standard starch sample for two hours in a convection oven set at the temperature determined by TGA in **4.4.1**. Cool the samples in a weighing bottle in a desiccator. Quickly transfer each of the dried standard starches to the TGA oven and run a TGA to determine whether all of the moisture has been removed. If no more moisture remains, drying for two hours at the temperature determined by TGA will be considered sufficient to dry the product.

If the TGA plot indicates that moisture still remains in the standard starch, dry for an additional two hours, cool as indicated above and rerun the TGA. Continue checking at two hour intervals until all of the moisture is removed. This new time (which may be different for each starch sample) should be used to dry all future starch standards.

4.4.3 Conduct Thermal Gravimetric Analysis (TGA) on each unknown sample to determine the temperature at which it loses moisture.

4.4.4 Dry, to the nearest 0.001 gram, 2 grams of each unknown sample for a specified time in a convection oven set at the temperature at which that particular sample lost its moisture determined by TGA in **4.4.3**. Cool the samples in a weighing bottle in a desiccator. Quickly transfer each of the dried samples to the TGA oven and run a TGA to determine whether all of the moisture has been removed. If no more moisture remains, drying for two hours at the temperature determined by TGA will be considered sufficient to dry the product. If the TGA plot indicates that moisture still remains in the unknown sample, dry for an additional two hours, cool as indicated above and rerun the TGA. Continue checking at two hour intervals until all of the moisture is removed. This new time (which will be different for each unknown) should be used to dry all future samples.

4.5 Determination of starch contents

4.5.1 Calculate the percent moisture in each of the standard starch samples using the following formula:

$$\% \text{ Moisture} = (A-B)/A \times 100$$

where A = Weight (grams) of undried sample
B = Weight (grams) of dried sample

4.5.2 Calculate the weight of starch, on a dry weight basis, in each of the standard samples using the following formula:

$$\text{Dry weight (in grams) of starch} = A - AC$$

where A = Weight (grams) of undried sample
C = % moisture determined in **4.5.1** for each of the samples

4.5.3 Plot a standard curve of Saccharimeter readings, taken immediately after

filtration, at one hour, four hours and after standing overnight, versus the dry weight of starch in each standard sample.

4.5.4 Calculate the percent moisture in each of the unknowns using the following formula:

% Moisture = (a - b)/a X 100

where a = Weight (grams) of undried sample
b = Weight (grams) of dried sample

4.5.5 Determine the dry weight "d" (grams) of starch in the unknown samples by reading off the standard curve for the particular starch of interest.

4.5.6 Calculate the percent starch on a dry weight basis for each of the unknown samples using the following formula:

% starch = d/(a - ac)

where a = Weight (grams) of undried sample
c = % moisture in unknown determined in **4.5.4** above
d = Weight (grams) read off the graph

5 STARCH DETERMINATION USING TRIFLUOROACETIC ACID (TFA)

5.1 Repeat **Part 4 - PROCEDURE**, using trifluoroacetic acid in place of hydrochloric acid.

5.2 Compare the reproducibility of the method using HCL and TFA.

6 CORRELATE SACCHARIMETER READINGS TO PERCENT STARCH

6.1 Plot the saccharimeter reading versus the percent starch for each different type of starch. From a slope of the line connecting the points, calculate the factor which can be used to multiply the Saccharimeter reading to obtain percent starch directly.

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 11-03 INDEX

AOAC 923.03
Ash of Flour
Direct Method

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

For certain products of Chapter 11 of the Harmonized Tariff of the United States (HTSUS) must not have an ash exceeded what is stated in Chapter Note 2. This method is used to determine the ash.

2 REFERENCES

AOAC 923.03
Ash of Flour
Direct Method

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 11-04

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AOAC 979.09
Protein in Grains
Kjeldahl Method

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

Grain and products are classified in Chapter 11 of the Harmonized Tariff of the United States (HTSUS). This method is used to determine the protein content in grain.

2 REFERENCES

AOAC 979.09
Protein in Grains
Kjeldahl Method

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 11-05

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AOAC 920.87
Total Protein in Flour

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

Products of the milling industry are classified in Chapter 11 of the Harmonized Tariff of the United States (HTSUS). This method is used to determine the protein content in flour.

2 REFERENCES

AOAC 920.87
Total Protein in Flour